

Heat damaged cement paste characterization using the nonlinear acoustic waves

YOUSFI Ismail^{1*}, LAFHAJ Zoubeir¹ and ARSON Chloé²

¹LML UMR CNRS 8107, Ecole Centrale de Lille, Villeneuve d'Ascq, France ; emails : yousfi_ept@yahoo.fr, zoubeir-lafhaj@ec-lille.fr

²School of Civil and Environmental Engineering, Georgia Institute of Technology, Atlanta, GA, USA ; email: chloe.arson@ce.gatech.edu

ABSTRACT

This paper focuses on the experimental characterization of the elastic properties of sound and heat-damaged cement paste for different water cement ratio. The method of the nonlinear acoustic is based on the Higher Harmonic Generation technique and allows measuring a non-linear elastic parameter (β). Experimental results show that the non-linear parameter grows exponentially with the temperature gradient fixed in the experiment. In this paper, an experimental set up, especially designed for this study, is proposed in order to evaluate the damage-induced by the non-linearities in the behavior of heat-damaged cement paste. An empirical law is proposed to relate β to the cement water content and to temperature.

INTRODUCTION

Nonlinear interaction of a monochromatic elastic wave should be a good tool for nondestructive evaluation of existing concrete cement based materials (Zumpano, G. and Meo, M.). Nonlinear indicators have already proved efficient in detecting global damage by exhibiting a significant sensitivity (Zumpano, G. and Meo, M.) regarding classical linear ultrasonic methods like wave speed or attenuation (Birks, A. S.). However, it is necessary to understand the influence of some parameters such as temperature or water saturation on the nonlinear processes. For this reason, an experimental study to characterize a heat damaged cement paste by the nonlinear acoustic waves (using Higher Harmonic Generation technique) (Lee, T. and Kyung, Y. J.) is treated and processed in this paper.

This study is divided into three parts. In the first part, the heat damage protocol is applied at cement paste sample's series, conditioned at different water cement ratio (0.3, 0.4 and 0.5). In the second part, the principles of the both techniques linear and nonlinear were detailed. Moreover the experimental procedure of the damage's detecting was described. In the last

part, the principle results obtained by both approaches were discussed. The influence of the water content and temperature on the modulus of Young and nonlinear parameter β was quantified (Jhang, K. Y.).

EXPERIMENTAL PROCEDURE: GENERATION OF HEAT DAMAGE

Cement Sample Preparation and Heat Treatment

In order to obtain various samples with different porosities, cement paste samples were made with three different water/cement (w/c) ratios: 0.3; 0.4 and 0.5. The samples are cylinders with a diameter of 37 mm and a height of 74 mm (Figure 1). They were cored from larger slabs of a Portland cement (CEM I 52.5N) preserved in limewater. Then they were rectified to obtain a parallel end-faces. Several heat treatment techniques were applied at cement paste in previous studies (Fu, Y.F. *et al.*, Xu, Y. *et al.*, Joshua, B *et al.*, Lion M. *et al.*). In this work, it was chosen to impose the temperature variations detailed and illustrated in Figure 2. The ambient case considered is the case of a saturated ambient temperature T_0 lying between 23°C and 30 °C (it was most often equal to 25°C in the laboratory). To prepare damaged cement, the sample was first put in the oven for 120 min, during which temperature was progressively raised up to a maximum temperature T_{max} . In order to avoid heat shocks, the temperature was increased incrementally from T_0 to T_{max} . The samples were then maintained at temperature T_{max} for 60 min. Then, samples were exposed to ambient temperature. Advection heat exchanges with the atmosphere made the sample temperature decrease. For each ratio w/c, heat damaged samples were studied for four maximum temperatures (80°C, 140°C, 200°C and 240°C), and compared to a sound sample. For this reason, five samples were prepared for each ratio w/c.

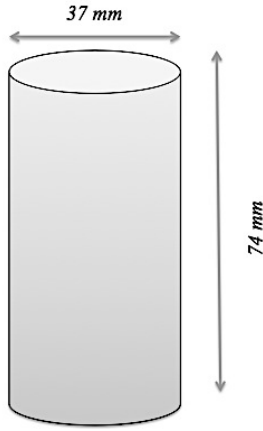


Figure 1. Dimensions of samples

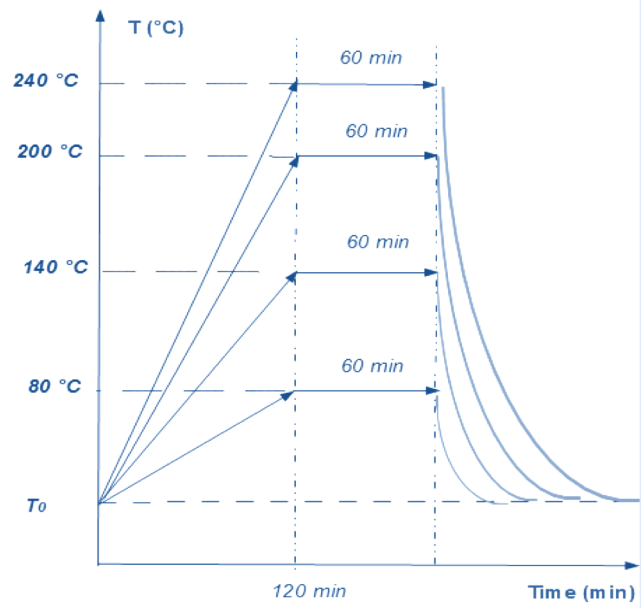


Figure 2. Time evolution of temperature during heat treatment

DETECTION OF DAMAGE BY ACOUSTIC METHODS

Linear Acoustic Method

Linear Acoustic Methods were used extensively at Ecole Centrale de Lille (ECL) in order to characterize the elastic and permeability properties of cement-based materials (Lafhaj, Z. and Goueygou, M., Goueygou, M. *et al.* , Lafhaj, Z. *et al.*). This technique allows measuring the velocities (Longitudinal and Shear Velocities) of pulse waves transmitted through a material sample. Assuming that the time-of-flight Δt (i.e. the time for the wave to cross the entire sample) can be measured in the laboratory, the wave velocity V is computed as:

$$V = \frac{L}{\Delta t} \quad (1)$$

in which L is the length of the sample.

Mechanical properties, Young's modulus E and Poisson's coefficient ν , are related to wave velocities according to equation 2 and equation 3 respectively :

$$E = \frac{\rho V_L^2 (3V_L^2 - 4V_T^2)}{(V_L^2 - V_T^2)} \quad (2)$$

$$\nu = \frac{1}{2} \frac{1 - 2\left(\frac{V_T^2}{V_L^2}\right)}{1 - \left(\frac{V_T^2}{V_L^2}\right)} \quad (3)$$

Where V_L and V_T are respectively longitudinal and shear velocities, and ρ is the mass density of the cement sample.

Non-Linear Acoustic Method

Actually, Cement paste is considered as an inhomogeneous micro-material that includes heterogeneities larger than the inter-atomic distance. From the other side, Acoustic methods are based on the measurement of wave velocities where, the wavelength is larger than the size of these irregularities. As a result, cement paste shows a non-linear behavior, even in the undamaged or sound state (this is typical of microscopic inhomogeneous materials). However, when the sample starts to deteriorate, the non-linearity turns into an entirely new dimension. This new non-linear mode tends to dominate the initial non-linear mode characterizing the sound material. Therefore, it measures the eventual level of damage including the smallest defects occurring in the early stages of heat treatment. So, as long as the damage-induced, non-linearity can be detected. In fact, Non-Linear Acoustic Methods permit such measurements, as opposed to the Linear Acoustic Method explained earlier. The technique of *Higher Harmonic Generation Method* requires sending a sine wave of frequency f in the cement paste sample (see Figure.3). Fourier Transform is then used to treat the received signal and express it in the frequency space. Spectral harmonics are obtained for multiples of the frequency $f : f, 2f, 3f, 4f \dots$ (see Figure 4). In this case, the two first amplitude peaks were determined in order to compute a non-linear parameter (β), which is related to the elastic stress/strain constitutive law. Actually β stems from the extension of Hooke's law to a second-order stress/strain relationship (Quan, L. *et al.*, Kyung-Young, J.):

$$\frac{\partial \sigma}{\partial \epsilon} = E(1 - \beta \epsilon) \quad (4)$$

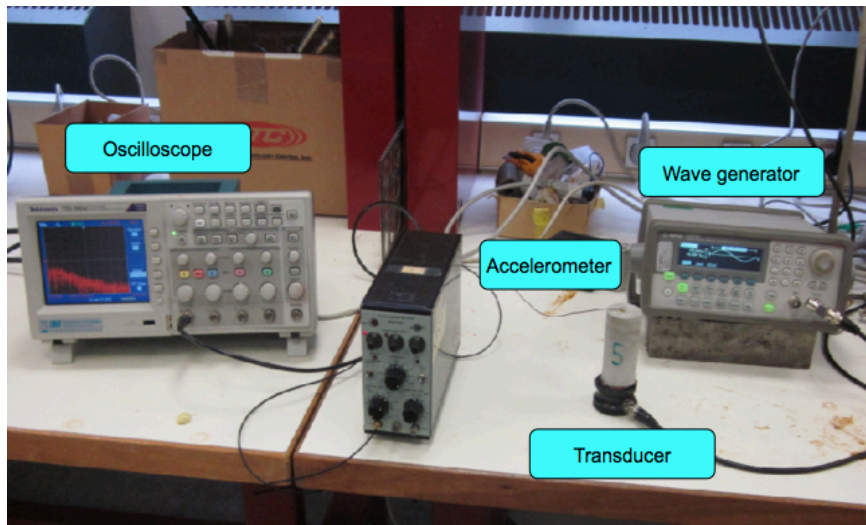


Figure 3. Experimental device used with the Non-Linear acoustic method.

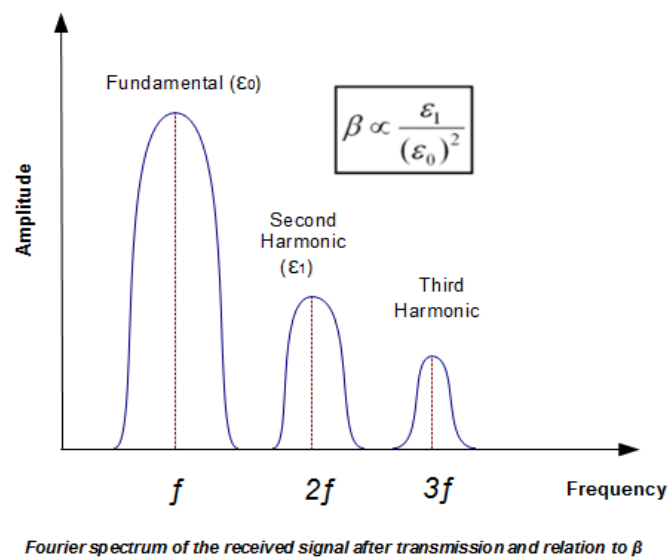


Figure 4. Principle of the High Harmonic Generation Technique.

RESULTS AND DISCUSSIONS

Linear Acoustic Method

The Equation 2 is used to deduce the Young modulus altered by heat treatment from wave velocity measurements. The Young moduli of sound samples are provided in Table 1 , and the evolution of cement paste Young modulus with temperature is plotted in Figure 5 for the three ratios w/c. Young's modulus decreases as temperature is raised, following a logarithmic curve. From the slope of the curve showing the evolution of Young's modulus with temperature, it can also be noted that higher w/c ratios tend to enhance the degradation process. As an example: for the same temperature 240°C, the normalized Young's modulus of the sample at ratio of w/c=0.5 is equal to 0.3, but for w/c=0.3, it is equal to 0.7, which is approximately the double value.

Table 1. Initial values of Young's modulus of the sound samples

w/c	0.3	0.4	0.5
Initial modulus of Young (GPa)	34.54	25.57	22.77

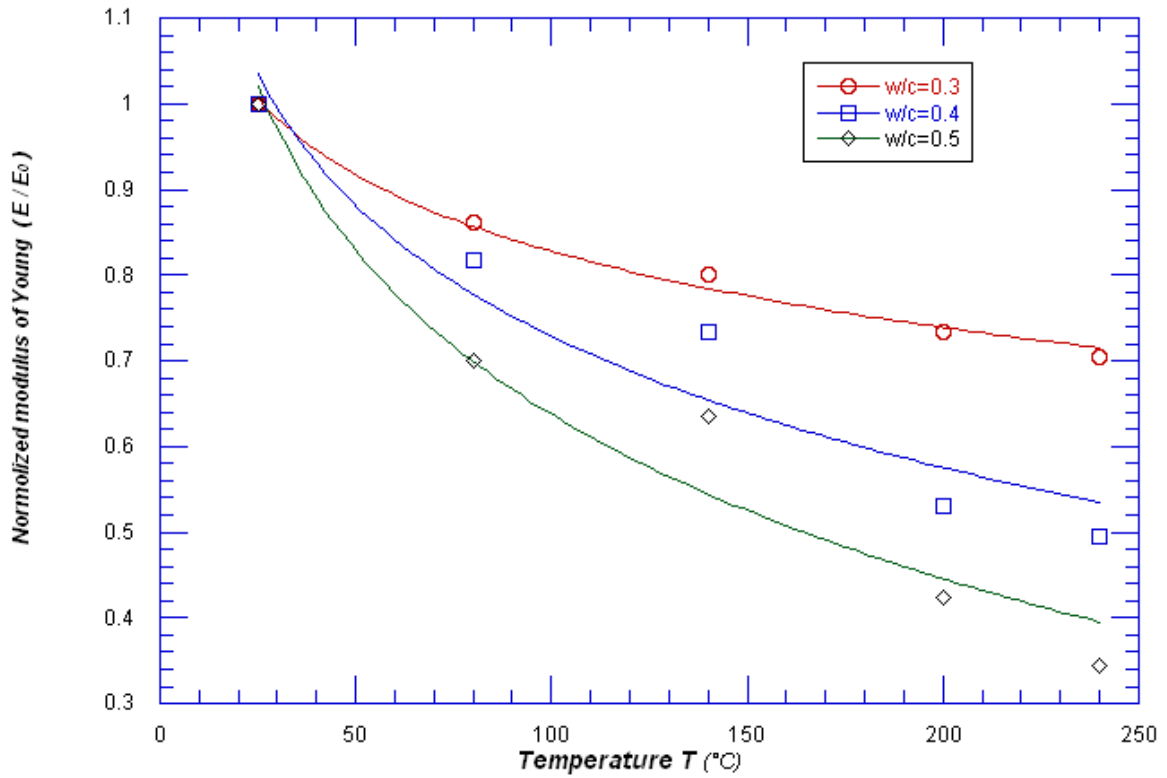


Figure. 5. Evolution of Modulus of Young with Temperature, as measured with the Linear Acoustic Method (logarithmic trend).

Non-Linear Acoustic Method

Figure. 6 displays the evolution of the nonlinear parameter β versus to temperature, for the three values of w/c under study ($w/c=0.3, 0.4, 0.5$). It can be observed that the nonlinear parameter is affected by both temperature and water cement ratio. For a given value of water content a significant increase of β with increasing temperature or damage degree. As an example, a climb of β , for $w/c=0.5$, is about 55% when temperature increases from 140°C to 240°C, yet this climb is about 18% when temperature rise from 25°C to 140°C. After plotting the trend curves an exponential function is found between the temperature and non linear parameter. This demonstrates the sensitivity of the stress/strain relationship in Equation 4 to the non-linear damage (i.e., to damage which cannot be represented by the simple relation given in Equation 4). The nonlinear parameter β is also sensitive to the w/c ratio, i.e. to porosity. An empirical formula is proposed, based on the fitting curve displayed in Figure .6:

$$\beta\left(\frac{w}{c}, T\right) = \frac{w/c + 0.1}{100} \exp\left(\frac{w/c}{100} T\right) \quad (5)$$

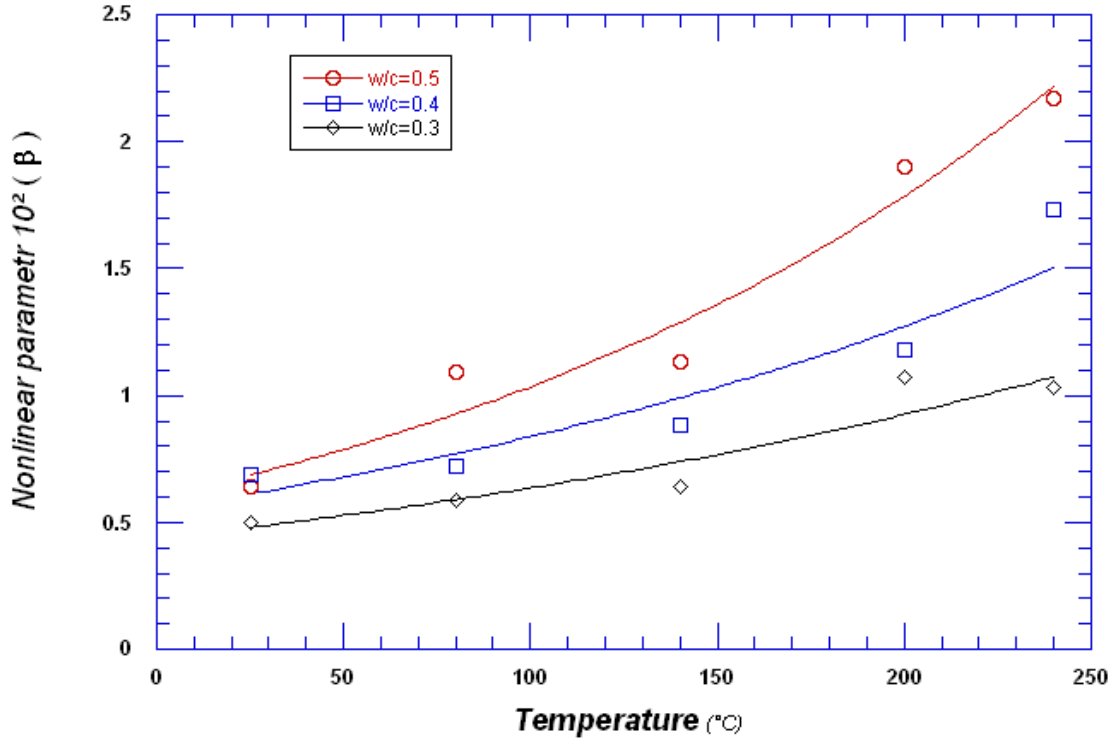


Figure. 6. Evolution of the Non-Linear Parameter β with Temperature (exponential trend).

CONCLUSION

Cement paste samples with different initial porosities were subjected to different temperature gradients in order to evaluate the effect of thermal damage on cement elastic properties, and to establish a relationship between damage, temperature and water content. In this paper, the Linear Acoustic Method allows relating wave velocities to Young's modulus and Poisson's ratio. The variations of Young's modulus with temperature changes show a logarithmic trend. Although the Linear Acoustic Method is easy and fast to implement, it is limited to damage models assuming that the sound material is linear elastic. This technique is not satisfactory for an inhomogeneous micro-material such as cement paste. For this reason, a technical device

was implemented in order to use a sophisticated damage assessment technique, based on the Higher Harmonic Generation Method. A non-linear parameter (β) is introduced in the constitutive law, and the Higher Harmonic Generation Method is used to measure β for the various temperature changes imposed as part of cement heat treatment. Experimental measurements provide evidence of an exponential growth of β with temperature gradient. An empirical law was provided. It relates β to temperature and cement water content. More work is needed to test the validity of this empirical model for a larger range of damage states, including crack coalescence. However, the present work is expected to give new insights in the field of Non Destructive Testing Methods. In particular, this preliminary study could be used to detect the non-linear behavior of standard civil engineering materials (such as concrete, mortar and cement) in the early stages of damage. Non Linear Acoustic Methods are expected to enable earlier failure diagnosis and thus reduce maintenance costs of infrastructure.

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